

United States Department of Agriculture
Agricultural Research Administration
Bureau of Entomology and Plant QuarantineINSECTICIDAL ACTION OF ORGANIC HALOGEN COMPOUNDS--
A COMPARISON OF SELECTED LITERATURE REFERENCESBy C. V. Bowen and H. L. Haller
Division of Insecticide Investigations

Interest in organic compounds containing halogens as insecticides has been stimulated recently by the widespread publicity given to DDT (1-trichloro-2,2-bis(p-chlorophenyl)ethane) (2, 15, 17); by the effectiveness as a soil disinfectant in pineapple fields of a mixture containing 1,3-dichloropropylene and 1,2-dichloropropane, the commercial product of which has been called D-D (5); and by 1,2,3,4,5,6-hexachlorocyclohexane, a new British insecticide (20, 1). Dichloropropane was first tested as an insecticide 20 years ago (13) and was shown to have limited value as a fumigant. DDT is of especial interest because of its high residual toxicity, and D-D because as a byproduct in the chlorination of hydrocarbons it is available at a comparatively low cost. 1,2,3,4,5,6-Hexachlorocyclohexane, or benzene hexachloride as it is commonly called, was discovered to have insecticidal properties in 1942, and early in 1943 the gamma isomer was reported to be more toxic to some insects than DDT.

D-D, DDT, and benzene hexachloride represent but a small percentage of the total number of halogen-containing compounds that have been tested against a wide variety of insects. Because of the great interest shown in these three products, it appeared worth while to assemble the toxicity data of some other halogen-containing compounds, and to observe the effect of the introduction of one or more halogens into an organic compound on its insecticidal properties. For this purpose publications containing the results of tests with a large number of organic halogen compounds ^{1/} were selected.

Grain Weevils

Neifert et al. (13) studied the fumigating action of more than 100 volatile organic compounds against grain weevils. Of the 34 organic halogen compounds, 9 were more toxic than carbon disulfide to the rice weevil (Sitophilus oryza (L.)). The most effective bromide tested was ethylene bromide, which at a concentration of only 0.5 percent killed 100 percent of the weevils (S. oryza (L.), S. granarius (L.), and Tribolium spp.). The order of toxicity of the other bromides was bromoform; n-butyl, ethyl, allyl, n-propyl, and benzyl bromides; and bromobenzene. Epichlorohydrin, trichloroethane, sym-tetrachloroethane, propylene dichloride, and

^{1/} The names of the chemical compounds are given as they appear in the literature cited.

a mixture of o- and p-dichlorobenzenes at concentrations of 2 percent or less gave 100 percent mortality. sym-Dichloromethyl ether had an efficiency comparable to that of carbon disulfide; chloromethyl ether was about half as efficient. Epichlorohydrin, the most toxic substance tested, at a concentration of only 0.09 percent killed 100 percent of S. oryza. Other chlorides showed toxicity in the following order: Monochlorobenzene, p-dichlorobenzene, tetrachloroethylene, methylene chloride, carbon tetrachloride, chloroform, acetylene dichloride, trichloroethylene, ethylidene chloride, and isopropyl chloride. The bromides in general were more effective than the corresponding chlorides. n-Butyl iodide, the only iodine compound tested, at a concentration of 0.8 percent killed all the weevils.

Roark and Cotton (16) reported further on the toxicity of 303 aliphatic halogen compounds tested as fumigants in the presence of wheat against the rice weevil. The most effective of these compounds, together with the minimum lethal dosages, are listed below.

<u>Milligrams</u> <u>per liter</u>		<u>Milligrams</u> <u>per liter</u>	
tert-Butyl bromide	<24	tert-Butyl chloride	34
Epichlorohydrin	<24	2-Chloroethyl acetate	47
2-Chloroethyl ether	<24	Carbon tetrabromide	60
2-Bromoethyl ethyl ether	<27	Isobutyl iodide	64
Allyl bromide	<28	sec-Butyl iodide	64
2-Bromoethyl acetate	<30	Isopropyl iodide	68
Methyl bromoacetate	<30	Methyl chloroacetate	73
Ethyl bromoacetate	<30	n-Propyl bromide	81
n-Propyl iodide	<35	Ethyl 1-bromopropionate	84
Allyl iodide	<37	Ethylene dibromide	87
Ethyl iodide	<39	Propylene chlorohydrin	89
Methyl iodide	<46	Ethyl chloroacetate	93
Methylene iodide	<67	n-Butyl iodide	97

The following 19 halogen compounds failed to kill any weevils at the maximum dosage of 0.50 ml. per liter: sym-Tetrabromoethane, 1,2,3-tri-bromobutane, hexachloroethane, iodoform, trimethylene chlorohydrin, chloromethyl ether, chloroacetic acid, bromoacetyl bromide, chloroacetyl chloride, dichloroacetyl chloride, trichloroacetyl chloride, propionyl chloride, n-butyryl chloride, isovaleryl chloride, n-butyl chloroformate, isoamyl chloroformate, gamma-bromopropyl acetate, chloral cyanohydrin, and n-butane-sulfonyl chloride. From the results presented by Roark and Cotton it is evident that in general the alkyl iodides tested are more toxic than the corresponding bromides and chlorides, and that the bromides are more toxic than the chlorides. Table 1 shows the effect of halogen substitution in some of the compounds tested. Although in some cases the toxicity was increased by the substitution of a halogen, in a number of others the toxicity was decreased.

Mosquitoes

Approximately 400 organic compounds were investigated by Fink et al. (8) to determine their toxicity to Culex mosquito larvae. Of these compounds 96 contained halogens. Of the 65 chlorine compounds 42 showed some toxicity; 18 of the 20 bromine compounds and 12 of the 14 iodine compounds were toxic to some degree. Their results with some of the compounds are shown in table 2 in comparison with those obtained with the parent compound. The introduction of 1 chlorine or bromine atom in biphenyl resulted in increased toxicity, the substitution of 2 chlorine atoms had the opposite effect, whereas the dibromo and diiodo derivatives were nontoxic. Chlorine substitution decreased the toxicity of dibenzofuran, had no effect on the toxicity of 2-nitrodibenzofuran and 1-phenylazo-2-naphthol, but increased the toxic effect of 4-nitroaniline and N-acetyl-diphenylamine.

Bushland and King (4) more recently have tested 113 organic compounds against mosquito larvae (Culex quinquefasciatus Say). The 24 halogen compounds gave mortalities in 16 hours ranging from 3 percent at a concentration of 100 p.p.m. to 99.5 percent at 1.25 p.p.m.

Table 3 shows the comparative toxicity of related compounds and results obtained by both groups of investigators on the same compound. No relation between toxicity and the halogen substituent is evident.

European Corn Borer

An investigation by Questel et al. (14) of the toxicity of 122 compounds to the European corn borer (Pyrausta nubilalis (Hbn.)) included 21 halogen compounds. Five of the 11 chlorine compounds tested were toxic, 2 of the 4 bromine compounds, and all 6 of the iodine compounds. Table 4 shows that in 4 of the compounds tested a halogen substitution resulted in a decrease in toxicity to the corn borer.

Screwworm

Bushland (3) reports from his tests of 551 organic compounds against screwworm larvae that "one hundred and seventeen compounds containing one or more halogen atoms were tested. Eighteen of these were of outstanding toxicity, but fourteen of these eighteen possessed a nitro group and one was a derivative of quinoline. Two of the compounds were derivatives of diphenyl (the p-chloro and p-bromo). The other halogen compound was beta-iodonaphthalene." The p-chlorodiphenyl was more toxic than diphenyl, while the p-iodo derivative was considerably less toxic. He further states, in regard to the diphenyl derivatives, that "the introduction of a chlorine atom in the para position of one of the benzene rings enhanced toxicity, but chlorine substituted in the ortho position reduced toxicity. When both benzene rings were substituted with halogen in the para positions, the resultant compounds (4,4'-dibromodiphenyl, 4,4'-dichlorodiphenyl) had little or no toxicity." The following 11 compounds were nontoxic at a concentration of 0.67 percent, although the parent compound showed some

toxicity: 4,4'-Dibromodiphenyl, 2,4-dibromo- α -naphthol, p,p'-dichloroazoxybenzene, 2,4-dichloro-1-naphthol, 2,6-dichloro-4-nitroaniline, 2-nitro-4-chloroaniline, 4-nitro-2-chloroaniline, 2,4,6-tribromophenol, 2,4,5-trichlorophenol, p-nitroiodobenzene, and 2,4,6-triiodophenol. 4,4'-Dichlorodiphenyl and p-dichlorobenzene possessed some toxicity, but the corresponding bromine compounds were nontoxic. A comparison of the toxicity of the biphenyl derivatives to different insects is presented in table 5.

Codling Moth

The toxicity of organic compounds to the larvae of the codling moth (*Carpocapsa pomonella* (L.)) is reported by McAlister and Van Leeuwen (12), and by Siegler et al. (18,19). The former rated the compounds according to their efficiency. The chlorine compounds had efficiencies from 0.0 to 98.7 percent, while the bromine compounds rated from 16.4 to 100 percent. No consistency in the toxicity of the halogen derivatives of barbituric acid, diphenylpiperazine, and nitrobenzene is evident from the following list:

	<u>Percent Efficiency</u>
Barbituric acid	16.4
Dibromobarbituric acid	65.6
Dichlorobarbituric acid	16.4
Diphenylpiperazine	80.3
Diphenylpiperazine hydrochloride	26.2
Nitrobenzene	58.2
m-Nitrobromobenzene	16.4
o-Nitrobromobenzene	31.2
p-Nitrobromobenzene	86.4
o-Nitrochlorobenzene	20.1
p-Nitrochlorobenzene	61.9

No iodine compounds were tested. Siegler, however, reported the percentage of wormy and stung apple plugs, from which, with the check value for his larvae, it may be determined whether a compound is toxic or nontoxic. Of the 27 chlorine compounds 15 were nontoxic, 5 of the 9 bromine compounds, and 6 of the 7 iodine compounds. Anthraquinone and α -furalacetophenone were toxic, but the respective 2-chloro and p-chloro derivatives were nontoxic. Biphenyl was toxic, as were also the p-chloro, p-bromo, and 4,4'-dichloro derivatives, but 4,4'-dibromo-, 4,4'-diiodo-, o-iodo-, and p-iododiphenyl were nontoxic, as shown in table 5.

Siegler et al. (18) also studied the toxicity of the halogenated benzenes, nitrobenzene, and the three isomeric monohalogenated nitrobenzenes, dihalogenated (except difluoro) benzenes, and dinitrobenzenes. The monohalogenated benzenes and the chloronitrobenzenes showed little toxicity to the codling moth larvae. In the bromonitrobenzene series, however, the para derivative was more toxic than the ortho or the meta compounds. All the

iodonitrobenzenes were toxic, the ortho least and the para most toxic. There was no significant difference in toxicity between the isomeric dihalogenated benzenes. The authors stated that "the data obtained indicate no marked correlation between either the groupings involved or their relative positions, with regard to their toxicity to the codling moth larva."

Wireworms

Tattersfield and Roberts (21), after a study of the influence of chemical constitution on the toxicity of organic compounds to wireworms (Agriotes spp.), found that the position of the halogen in the aromatic ring was practically without effect, and noted a consistent increase in toxicity on the first chlorine substitution but a slightly greater effect on the second chlorine substitution. The toxicities of related compounds, measured in millionths of a gram-molecule per 1000 cc. of air found toxic in 1000 minutes at 15° C., are given below. The first figure represents the death point, and the second figure the recovery point.

Chloroform	1040-800
Bromoform	94
Iodoform	Nontoxic
Carbon tetrachloride	1600
Chlorobenzene	200-170
<u>o</u> -Dichlorobenzene	70-50
<u>p</u> -Dichlorobenzene	Marginal
1,2,4-Trichlorobenzene	Marginal
Bromobenzene	96-80
Iodobenzene	50-25

Fabric Insects

Tests against the webbing clothes moth (Tineola bisselliella (Hum.)) and the furniture carpet beetle (Anthrenus vorax Waterh.) made by Colman (6) with 165 organic compounds, 48 of which contained halogens, gave no evidence of a relationship between the toxicity and the halogen substitution. In the following list the lowest ratio between the damage caused by partly grown larvae of the carpet beetle to cloth treated with the test material and to cloth treated with a standard mothproofing agent (sodium fluosilicate) indicates the most toxic compound.

	Ratio
Acetophenone semicarbazone	4.1
<u>p</u> -Chloroacetophenone semicarbazone	1.4
3,4-Dichloroacetophenone semicarbazone	4.2
Benzaldehyde semicarbazone	4.6
<u>o</u> -Chlorobenzaldehyde semicarbazone	10.4

The o-chloro-, p-chloro-, 2,4-dichloro-, and 2,4,6-tribromo- phenyl esters of p-toluenesulfonic acid are rated by Colman as having the same toxicity as the phenyl ester of p-toluenesulfonic acid when tested against newly hatched larvae of the webbing clothes moth. With the exception of m-chloro-

acetanilide, which gave 100 percent mortality of newly hatched larvae but not of 6-weeks-old larvae, no difference was noted in the results obtained with the ortho, meta, and para isomers of the chloro-, bromo-, and iodoacetanilides.

Cupples, Yust, and Hiley (7) reported on tests with more than 300 compounds as possible substitutes for hydrocyanic acid in the fumigation of California red scale (*Aonidiella aurantii* (Mask.)). Of the 89 organic halogen compounds, 79 showed only slight or no toxicity, 7 moderate toxicity, and 3 decided toxicity. Twenty-four cases of related compounds showing the relative toxicity with change in halogen content are indicated as follows:

<u>n</u> -Octane	= 2-Bromooctane
Cyclohexane	= Bromocyclohexane
Benzene	= <u>p</u> -Dichlorobenzene
Methylene chloride	> Chloroform = Carbon tetrachloride
Methyl bromide	= Methylene bromide < Bromoform
<u>sym</u> -Dichloroethylene	< Trichloroethylene > Tetrachloroethylene
Fluorobenzene	= <u>p</u> -Fluorochlorobenzene
	= <u>p</u> -Fluorobromobenzene
Benzyl chloride	= Benzotrichloride
	= <u>o</u> -Bromobenzyl chloride
Ether	= <u>beta</u> -Bromoethyl ethyl ether
Nitromethane	< Chloropicrin
	< Bromopicrin
Ethyl acetate	< Ethyl bromoacetate
	= <u>beta</u> -Bromoethyl acetate
<u>n</u> -Propyl acetate	= <u>beta</u> -Bromopropyl acetate
	= <u>gamma</u> -Bromopropyl acetate
Benzyl acetate	= Benzyl chloroacetate

Fourteen compounds showed no change in toxicity with increase of halogen content, three showed a decrease, and six an increase in toxicity.

Goldfish

In toxicity tests of halogenated phenols against goldfish, Gersdorff and Smith (9, 10, 11) found that the most pronounced differences between the iodophenols and the chloro- and bromophenols are as follows: (1) Each iodophenol is more toxic than the corresponding compound containing chlorine or bromine. (2) The ortho and meta compounds have changed places, however, in the order of toxicity so that the latter is the least toxic of the iodophenols. (3) The para compound has a pronouncedly greater toxicity than its least toxic isomer, it being five times as toxic in the case of the iodophenols as compared with one and one-half times for the bromo- and chloro- phenols.

Conclusions

The dissimilarity of treatment and the wide variety of compounds

tested in the entomological investigations reported make the drawing of conclusions difficult. It is apparent, however, that the introduction of halogen into an organic compound may increase, decrease, or have no effect on the insecticidal value, and that the effect on toxicity obtained by the introduction of one of the halogens is no indication of the effect likely to be obtained by the introduction of a different halogen.

Table 1.--Effect of halogen substitution on effectiveness of compounds as fumigants for wheat against the rice weevil

Compound	Minimum lethal dosage	Weevils killed after 24 hours
	Milligrams per liter	Percent
Methyl formate	39	100
Methyl chloroformate	198	100
Ethyl formate	72	100
Ethyl chloroformate	251	100
Chloroethyl chloroformate	480	100
n-Propyl formate	72	100
n-Propyl chloroformate	542	20
gamma-Chloropropyl chloroformate	600	10
Isopropyl formate	53	100
Isopropyl chloroformate	540	70
n-Butyl formate	109	100
n-Butyl chloroformate	539	0
Isobutyl formate	35	100
Isobutyl chloroformate	520	20
Isoamyl formate	70	100
Isoamyl chloroformate	512	0
Chloromethyl ether	532	0
sym-Dichloromethyl ether	658	100
Diethyl ether	357	10
beta-Bromoethyl ethyl ether	27	100
alpha,beta-Dichloroethyl ether	188	100
beta,beta-Dichloroethyl ether	24	100
Acetone	396	100
Chloroacetone	110	100
Acetic acid	500	0
Chloroacetic acid	496	0
Chloroacetyl chloride	748	0
Dichloroacetyl chloride	780	0
Trichloroacetyl chloride	815	0
Ethyl acetate	180	100
2-Bromoethyl acetate	30	100
2-Chloroethyl acetate	47	100
Ethyl bromoacetate	30	100
Ethyl chloroacetate	93	100
Ethyl dichloroacetate	256	100
Ethyl trichloroacetate	692	100
Ethyl alcohol	790	40
Ethylene chlorohydrin	242	100
Ethylene bromohydrin	337	100
n-Propyl alcohol	402	60
Propylene chlorohydrin	89	100

Table 2.--Toxicity tests against mosquito larvae, showing parent compound and halogen derivatives

Compound	Concentration	Mortality in 16 hours	Toxicity compared with parent
	P.p.m.	Percent	
Biphenyl	10	54	
p-Bromobiphenyl	3.3	51	V
p-Chlorobiphenyl	5	84	V V
p-Iodobiphenyl	10	66	V
4,4'-Dichlorobiphenyl	100	65	A A A
4,4'-Dibromobiphenyl	100	0	A A A
4,4'-Diiodobiphenyl	100	0	A A A
Dibenzofuran	5	80	
2-Chlorodibenzofuran	5	50	A
2-Nitrodibenzofuran	100	0	
3-Chloro-7-nitrodibenzofuran	100	0	"
p-Nitroaniline	100	54	
2-Chloro-4-nitroaniline	20	98	V
N-Acetyldiphenylamine	100	0	
N-Chloroacetyldiphenylamine	100	43	V
1-Phenylazo-2-naphthol	100	0	
1-(2,5-Dichlorophenylazo)-2-naphthol	100	0	"
Phenetole 1/	100	41	
o-Bromophenetole 1/	20	66	V
p-Bromophenetole 1/	20	94	V
Anisole 1/	100	22	
o-Bromoanisole 1/	50	73	V
p-Bromoanisole 1/	50	92	V
Hydrazobenzene	10	92	
p-Bromohydrazobenzene 1/	1	93	V
Acenaphthene	20	94	
3-Chloroacenaphthene 1/	20	96	"

1/ Data from Bushland and King (4).

Table 3.-- Toxicity tests of related compounds against mosquito larvae
(Data from Fink et al. (8) unless otherwise stated)

Compound	Concentration	Average mortality	
		: After 16 hours	: After 40 hours
	P.p.m.	Percent	Percent
Hexachloroethane	30	87	-
<u>o</u> -Dichlorobenzene <u>1/</u>	<u>1/</u> 20	95	97
<u>p</u> -Dichlorobenzene	<u>1/</u> 50	99	99
	<u>1/</u> 50	98	96
	40	95	-
<u>p</u> -Dibromobenzene	20	52	-
<u>p</u> -Diiodobenzene	40	58	-
<u>m</u> -Chloronitrobenzene	100	100	-
<u>o</u> -Chloronitrobenzene	100	100	-
<u>p</u> -Chloronitrobenzene	40	73	-
3,4-Dichloronitrobenzene <u>1/</u>	20	85	91
<u>m</u> -Bromonitrobenzene	40	96	-
<u>o</u> -Bromonitrobenzene	40	64	-
<u>p</u> -Bromonitrobenzene	5	48	-
<u>m</u> -Iodonitrobenzene	20	96	-
<u>o</u> -Iodonitrobenzene	40	98	-
<u>p</u> -Iodonitrobenzene	100	0	-
<u>p</u> -Iodosonitrobenzene	<u>1/</u> 100	5	5
	100	0	-
<u>m</u> -Nitrophenyliodochloride <u>1/</u>	<u>1/</u> 20	18	20
<u>p</u> -Nitrophenyliodochloride <u>1/</u>	<u>1/</u> 100	7	36
<u>p</u> -Thiocyanobromobenzene <u>1/</u>	<u>1/</u> 2	97	96
<u>p</u> -Thiocyaniodobenzene <u>1/</u>	<u>1/</u> 2	98	98
2,4-Dinitrochlorobenzene <u>1/</u>	<u>1/</u> 20	96	99
2,4-Dinitrobromobenzene <u>1/</u>	<u>1/</u> 20	98	99

1/ Data from Bushland and King (4).

Table 4.-- Effect of halogen substitution on toxicity of some organic compounds to the European corn borer

Compound	: Mortality in laboratory tests				: Reduction in borers			
	: 4 lb. per 100 gal. : 1 lb. per 100 gal.				: over untreated plants			
	: Treated: Untreated : Treated: Untreated				: in field tests			
	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent
Acetophenone semicarbazone	98.8	5.8	20.0	8.7	--	--	--	--
p-Chloroacetophenone semicarbazone	91.2	2.3	2.0	4.5	--	--	--	--
3,4-Dichloroacetophenone semicarbazone	2.4	1.5	--	--	--	--	--	--
Benzaldehyde semicarbazone	94.2	5.8	--	--	--	--	--	--
o-Chlorobenzaldehyde semicarbazone	2.0	.9	--	--	--	--	--	--
Fluorene	98.3	2.0	100	4.5	43.7	--	--	--
2-Chlorofluorene	--	--	99.3	3.0	67.9	--	--	--
Azobenzene	--	--	--	--	--	59.1	--	--
p-Bromoazobenzene	100	2.0	100	4.5	46.0	--	--	--
p-Iodoazobenzene	100	2.0	79.0	4.5	49.1	--	--	--
Azoxybenzene	98.2	8.3	60.0	8.3	--	--	--	--
p,p'-Diodoazoxybenzene	36.8	10.7	--	--	--	--	--	--

Table 5.-- Effect on toxicity of halogen substitution in biphenyl

Compound	: Codling moth larvae 1/ :		: Mosquito larvae 2/ :		: Screwworm 3/ :	
	: Concentration : Wormy: Stung :		: Mortality : in 16 hours :		: Minimum lethal concentration :	
	Lb. per 100 gal.	Percent	Percent	P.P.M.	Percent	Percent
Biphenyl (diphenyl)	2	53	0	10	54	0.05-0.08
o-Bromobiphenyl	2	91	4	--	--	--
p-Bromobiphenyl	4	27	32	3.3	51	0.05-0.08
o-Chlorobiphenyl	--	--	--	--	--	0.33-0.67
p-Chlorobiphenyl	4	67	4	5	84	0.03-0.05
4,4'-Dibromobiphenyl	4	100	0	100	0	Nontoxic
4,4'-Dichlorobiphenyl	4	66	3	100	65	Slightly toxic 4/
4,4'-Diodobiphenyl	4	98	0	100	0	--
o-Iodobiphenyl	4	96	2	--	--	0.10-0.17
p-Iodobiphenyl	4	93	2	10	66	0.10-0.17

1/ Slegler et al. (19).

2/ Fink et al. (8).

3/ Bushland (3).

4/ Affected the larvae adversely but did not cause 100% mortality at a concentration of 0.67%.

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